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PATENT OF INVENTION**

**No. 76 15809**

(54) Process and installation for the production of fish protein concentrates.

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The invention covers a process for the manufacture of fish protein concentrates by enzymatic means, the enzymes used for the processing being the known proteolytic enzymes contained in the viscera of the fresh fish that constitute the raw material

The invention also covers an installation for implementing this process.

Different processes for the manufacture of fish protein concentrates are currently known. Among them we can cite, as an example, processes involving acid hydrolysis of the raw material or processes involving hot hydrolysis at high temperatures on the order of 100°C. Hydrolysis of the raw material consisting of the fish, when it is done under those conditions, can however entail the destruction of original vitamins as well as the destruction of an important part of the amino acids that comprise the total fish proteins.

It has therefore been proposed to produce such fish protein concentrates by enzymatic means, the enzymes necessary for this treatment being found in the viscera of the fish that constitute the raw material. However, the known processes using enzymatic treatment do not make it possible to obtain an optimum concentration of the original vitamins and amino acids.

One objective of the invention is to furnish a process for manufacturing fish protein concentrates by enzymatic means using the proteolytic enzymes contained in fish viscera, a process that makes it possible to obtain a fish protein concentrate in which the natural scale of the amino acids comprising all these proteins is precisely respected and a maximum concentration of original vitamins is obtained.

This goal is attained, according to the invention by a process for the manufacture of protein concentrates by enzymatic means in which the raw material consisting of fish and/or fish viscera, plus a certain amount of water, is quickly brought up to a temperature of around 40°C. The pH of the medium is then raised to around 4.5, possibly through the addition of acid, and the mixture is then being slowly brought to a temperature not to exceed 70°C for an amount of time such that a predetermined fraction of proteins is dissolved, these three phases constituting the digestion per se, and the liquid obtained at the conclusion of this digestion, which liquid consists of fish oil, a proteolysis juice rich in amino acids and sediments consisting of undissolved substances as well as undigested proteins, is then treated in an appropriate separate installation.

The purpose of the water added to the raw material is to facilitate hydrolysis, and it is generally added in a volume representing one-fourth the volume of the raw material.

The pH conditions of the medium must be determined and adjusted precisely if one wishes to obtain a protein concentrate with high nutritional value. The reaction medium heated to 40°C can be adjusted to a pH of around 4.5 by adding any acid that respects the natural scale of amino acids, for example hydrochloric acid, acetic acid, lactic acid.

The rapid rise in temperature to 40°C is generally accomplished in about 30 to 45 minutes. The second digestion phase during which the temperature is slowly raised to around 70°C lasts approximately 4 to 10 hours, the digestion or proteolysis time being directly proportional to the quantity of viscera compared to flesh of the fish comprising the raw material.

Whereas the pH remains at around 5 during the entire phase of slow rise in temperature, in other words, while the temperature rises from 40°C to around

70°C, the pH increases to a level of around 6 when the desired proportion of ichthyological proteins is dissolved.

A very simple means is therefore available for terminating the proteolysis, which is stopped when the pH of the reaction medium is around 6.

Another way of terminating the proteolysis consists in measuring the percentage of undissolved substances using a sample of the reaction medium which is subjected to centrifuging, then terminating the proteolysis operation when, after centrifuging, a quantity of dry substance is obtained that is less than a preset value, for example 25%, this dry material consisting of various undissolved substances such as scales, bones, etc., and of the proteins not digested during the enzymatic treatment.

An installation for implementing the process mainly includes, in addition to a digester, means for treating the liquid obtained at the end of the digestion or proteolysis and that still remains at a temperature in the neighborhood of 70°C. These means, capable of doing so-called "cascading" separations of the liquid in order to separate out its constituents, in other words the fish oil and fat, the proteolysis juice and sediments, including a first centrifugal separator that separates the sediments from a liquid consisting of the fish oils and fats and the proteolysis juice, a second centrifugal separator in which this liquid is introduced and which separates the fish oils and fats from the proteolysis juice, at least one device for treating the proteolysis juice which substantially reduces in water content, and a device for treating the sediments. The proteolysis juice obtained at the outlet of the second centrifuge separator can then be brought through a first branch conveyor to the proteolysis treatment device or devices and/or through a second branch conveyor to the intake of the sediment treatment apparatus.

The protein concentrates obtained pursuant to the invention can be used in human and animal nutrition because of other stimulating properties and their effects on asthenia reduction, weight gain and fatigue reduction.

These types of products can also be used in cosmetology.

The proteolysis juice also represents an interesting product because it is very rich in amino acids and can be subjected by any appropriate known process to treatment for separating the various amino acids that it contains, which amino acids can be used in the pharmaceutical industry.

The invention will be better understood from a reading of the following description, provided by way of example, and with reference to the attached drawing, in which:

Figure 1 is a diagram showing the different stages of an installation according to the invention; and

Figure 2 is a view in perspective of a form of embodiment of a digester used for the enzymatic treatment according to the invention.

The production of fish protein concentrates can be carried out in an installation of the type shown in Figure 1. In such an installation, the raw material consisting of fresh fish and/or the viscera of fresh fish is introduced into a grinder 10 where it undergoes a rough grinding. The grinder outlet is connected by a pipe or tube 11 to the intake of a digester 12 where the raw material, with a volume of water added representing about one fourth of its volume, undergoes the enzymatic treatment described earlier: the mixture is quickly brought to a temperature of around 40°C, the pH then being adjusted to around 4.5 and the temperature slowly raised to a maximum of 70°C, this second temperature increase phase continuing until a predetermined quantity of fish proteins has been dissolved.

The digesters used can be of two types, either horizontal or vertical, and are constructed with double walls, preferably made of stainless steel, to permit heating by bain-marie. A rotary or alternating agitator mounted in the digester is operated in such a way as to slowly shake up the mass to be digested, which makes it possible to distribute the heat and the active enzymes homogeneously throughout the entire mass.

Figure 2 shows a particularly advantageous form of embodiment of a digester in which the enzymatic treatment according to the invention can be done. This digester includes a stainless steel vat 110, substantially semi-cylindrical in shape, in which the material to be digested is dumped, and which is then supported by a second stainless steel vat 111, also substantially semi-cylindrical in shape, but of larger size, in such a way as to create a space between the walls of the two vats that is filled with the quantity of liquid, for example water, required for heating the mass contained in vat 110.

The heating liquid is itself heated by a coil 121 with expanded steam circulation, which coil is supported on the inside wall of the vat 111 by any appropriate means, for example by means of brackets or attachment lugs affixed to the inside wall of the vat.

The vat 110 is equipped with an agitator 112 rotary-mounted in the bearings 113 and 114 fixed to the front edges 115 and 116 of the vat 110. This agitator consists of a rod 117 to which is affixed a blade 118 whose length and width are slightly less than the length and radius of the semi-cylindrical vat 110, respectively; when in operation, this blade plunges into the mass contained in vat 110. The rod 117 is driven by an electric motor 122, a device 123 being interposed between the motor 122 and the rod 117 so as to transform the rotation movement provided by the motor into an alternating rotation movement. The motor is regulated in such a way that, during the digestion, an alternative rotation movement is imparted

to the rod 117 rotary-mounted in bearings 113 and 114, causing the blade 118 to do twenty to twenty-five beats a minute.

The motor 122 and the device 123 are supported by a bracket 124 affixed to the rear front face of the vat 111. This bracket also includes the equipment for regulating the temperature of the heating fluid as well as the necessary control equipment, for example, the pH control equipment. This type of equipment is known and is not shown.

A discharge pipe or tube 120 is installed on the front face 119 of the vat 110 and makes it possible to transfer the liquid obtained after digestion into the installation's separation apparatus.

When the production of fish proteins is done by implementing the process according to the invention, in other words, when the digestion of the reaction medium, first raised to 40°C then to 70°C after the pH of the medium has been adjusted to 4.5 is continued until the pH of the reaction medium has attained a value of around 6, the digestion being then interrupted by stopping the heating process and removing the reaction medium from the digester through a pump 13 and conveying it to a first centrifugal 14 through a pipe or tube 15.

The liquid obtained on output from the digester 12 and before treatment in the centrifugal separator 14 has substantially the composition indicated in Table 1 below, the percentages indicated being by weight:

Table 1

Fish oils and fats	8 to 15%
Proteolysis juice	50 to 70%
Sediments	20 to 45%

The centrifugal separator into which this liquid is introduced when it is still at a temperature of around 70°C thus makes it possible to separate the sediments (undissolved materials, undigested proteins) from the liquid portion consisting of fish oils and fats and proteolysis juice. This first centrifugal separator advantageously rotates with an acceleration of between 2,000 and 2,200 G.

The liquid portion is removed from the centrifugal separator 14 through a pipe 15' that conveys it to a second centrifugal separator 16 which in turn rotates at an acceleration advantageously ranging between 15,000 and 18,000 G. This second centrifugal separator separates the fish oil and fat from the proteolysis juice. The oils and fats, after decanting and possible filtration, are stored to be sold as is, whereas the proteolysis juice can either be utilized in its totality to enrich the sediments in amino acids and vitamins, or used in part to enrich these sediments, the remaining portion being treated to furnish protein concentrates, or also treated as a whole to furnish protein concentrates.

In the first case, the proteolysis juice is conveyed through a pipe 18 and a pump 19, from the centrifugal separator 16 to a rotary drier 17 that will be described below.

In the second case, part of the proteolysis juice follows the same path as above, in other words, it passes into the pipe 18 then, through pump 19, is conveyed into the rotary drier 17, and the remaining portion of the proteolysis juice is conveyed through a pipe 20 in which a filtration device may be installed, into various devices that will reduce the water content in the proteolysis juice and therefore ensure the preservation of the protein concentrates obtained.

This latter path is followed by all the proteolysis juice in the third case.



From the pipe 20, the proteolysis juice can be conveyed through a pipe 21 into a spray device 22, which treats the proteolysis juice in order to produce a protein concentrate in the form of a soluble powder.

From the pipe 20, the proteolysis juice can also be conveyed to a pipe 23 that conveys it to an evaporator 24, in which a large part of the proteolysis juice water is eliminated in order to obtain a protein concentrate.

The proteolysis juice can also be conveyed into any other appropriate device that makes it possible to reduce its water content, for example a vacuum concentrator, a freeze-drier, etc.

The proteolysis juice conveyed by the pipe 20 can finally be conveyed through a pipe 24 into a pasteurizer 26 in which it undergoes a treatment that makes it possible to preserve the product obtained for a long time before submitting it to further appropriate treatment. Such further treatment can be done, for example, in a spray device or in an evaporator and, in this case, all or part of the product obtained at the outlet 17 of the pasteurizer 26 is conveyed, either through a pipe 28 and a bypass 29 into the spray unit 22 either through the pipe 28 and a bypass 30 into the evaporator 24. The product obtained after treatment of the proteolysis juice in the pasteurizer 26 can also be removed through a pipe 36 and sent to any other appropriate transformation device.

The protein powders and concentrates obtained from the proteolysis juice are then conditioned in machines that are known in themselves and are not shown.

The sediments separated from the reaction mix by the centrifugal separator 14 are transported through a pipe 31 into the rotary drier 17 in which, after the addition, or not, of proteolysis juice as described above, they will lose a large part

of their water. This treatment is done through the quick passage of the sediments, possibly with proteolysis juice added, into the rotary drier 17, which is raised to a temperature of around 130-140°C, such passage preferably lasting only a few seconds.

The product obtained on output from the rotary drier 17 is then cooled very rapidly in a cooler 32. Thus, thanks to the fact that, on the one hand, the drying can only be continued for a few seconds because of the evaporation of the water, which takes place very quickly at this temperature, thus permitting the product not to exceed 80°C and that, on the other hand, the product obtained is then cooled very quickly, there is practically no deterioration of the proteins and amino acids. The product obtained after cooling in a cooler 32 is ground up in a grinder 33, screened in a screening device 34 and bagged in a bagging machine 35.

Such installation is preferably set up in an onshore plant. In such installation, which includes six to eight digesters with a volume of about 1.5 m<sup>3</sup> each, a first common centrifugal separator and a second common centrifuge, a team working an eight-hour shift can process up to ten tons of fish, which makes it possible to process a total of up to thirty tons of fish a day.

An installation to produce a fish protein concentrate by the process according to the invention can also be installed aboard a deep-sea fishing boat or a factory ship where the treatment is carried out on the fish immediately after they are caught. In this case, and in order to limit to the maximum extent possible the size of such on-board installation, the sediments, instead of being processed in a rotary drier, are subjected to vacuum evaporation or other treatment that makes it possible to reduce their water content and ensure their preservation for subsequent transformation by drying in an onshore plant.

The process according to the invention can also be applied to small production operations and in that case, a semi-automatic centrifuge working at around 4,000 G is used to successively separate the oil, the proteolysis juice and the sediments, the latter remaining in the centrifuge and being removed by hand. In this case, the proteolysis juice obtained must be filtered more carefully than in an installation using two centrifugal separators as described earlier, by filtration over clothe.

By way of example, tests involving the production of fish protein concentrates using the process according to the invention are reported below:

#### EXAMPLE 1:

150 kg of whole sardines and 100 kg of sardine viscera are rough-ground and 60 kg of water are added to the product of the grinding, the mixing being done in a digester like that shown in Figure 2. The temperature is then quickly raised to 40°C, the pH of the mixture then being 6.5. The pH of the reaction medium is then raised to 4.5 by the addition of 1.8 kg of around 10 N hydrochloric acid (20-21 degrees Baumé). The medium, constantly stirred in a digester of the type shown in Figure 2, is then gradually heated by adjusting the temperature of the bain-marie, making it possible to heat the vat containing the medium in order to obtain a temperature of 70°C after a total proteolysis time of 5 hours and 45 minutes.

The liquid obtained after digestion is then treated first in the first centrifugal separator 14, then in the second centrifugal separator 16 of the installation according to Figure 1, and the following is obtained:

Sediments:	82.4 kg	30.3% by weight
Oil:	26.9 kg	9.9% by weight
Proteolysis juice:	162.7 kg	59.8% by weight

25% of the proteolysis juice obtained, or around 40 kg, is treated in a vacuum concentrator where it loses a large part of its water : 7.8 kg of a fish protein concentrate containing 60% of dry extract is obtained.

The remaining fraction of the proteolysis juice, or 122.7 kg, is mixed with the sediments to enrich them in proteins and amino acids. The mixture thus obtained is treated in a rotary drier such as the one of the installation according to Figure 1 and, after cooling, grinding, screening and bagging, 47.4kg of dry powder is obtained.

#### EXAMPLE 2:

170 kg of whole mackerel and 80 kg of mackerel viscera are rough-ground, then transferred to a digester where they are mixed with 60 kg of water, the mixing being done in a digester such as the one shown in Figure 2. The temperature is quickly raised to 40°C, the pH of the medium then being 6. The pH of the medium is then brought down to 4.5 by the addition of 2 kg of around 10 N hydrochloric acid. The medium, which is continuously stirred in the digester, is gradually heated by adjusting the temperature of the bain-marie until, after a total proteolysis time of 6 hours 20 minutes, a temperature of around 70°C is obtained.

After digestion, the different ingredients of the reaction medium are first separated in the centrifugal separator 14, then in the centrifugal separator 16 of the installation according to Figure 1, with the following results:

Sediments:	54 kg	20% by weight
Oil:	36 kg	13.6% by weight
Proteolysis juice:	170 kg	66.4% by weight

25% of the proteolysis juice obtained, or 42.5 kg, is treated in a vacuum concentrator where it loses most of its water: thus, 8.9 kg of fish protein concentrate is obtained, concentrated to 60% dry extract.

The remaining fraction of proteolysis juice, or 127.5 kg, is added to the sediments to enrich them in proteins and amino acids, the mixture obtained being treated in a rotary drier, then in cooling, grinding, screening and bagging units like those of the installation according to Figure 1: this results in 43.7 kg of dry powder.

The level of proteins and other constituents or products of interest for human and animal nutrition was determined at two stages of the process according to the invention on raw material consisting of sardines and mackerel. The results of the analysis of the liquid collected on outlet from the digester, on the one hand, and on the other, and of the proteolysis juice after centrifuging are as follows:

Table 2

	Digester outlet % by weight	After centrifuging % by weight
Moisture	75 to 83	68 to 77
Dry substances	17 to 25	23 to 32
Mineral substances	5 to 3.4	4.2 to 4.8
Untreated proteins	4.75 to 5.25	6.5 to 9.5
Proteins on dry	22 to 25	28 to 30
Phosphorus	0.100 to 0.110	0.140 to 0.160
Calcium	0.130 to 0.195	0.200 to 0.250

Free aminated nitrogen	0.58 to 0.86	0.95 to 1
Oil	9.9 to 13.6	0

These results show that after centrifuging, the proteolysis juice contains no more oil and its extremely rich, especially in proteins.

The fish protein concentrates obtained according to the invention have an interesting application for human and animal nutrition.

These protein concentrates were fed to human beings and to various animals and the following observations were made.

#### TEST 1:

A protein concentrate obtained by the process according to the invention is mixed in a ratio of 15% by weight to skim milk. The product obtained was spray-dried and administered by mouth after dilution in ten times its volume of water in doses of 15 g/day to children suffering from Kwashiorkor, for 28 consecutive days. The quick disappearance of edemas and diarrhea was observed, along with a notable improvement in their weight curve.

#### TEST 2:

Protein concentrates manufactured according to the invention were administered to young rats during their breast-feeding period, as a supplement to the products usually administered to them, in a ratio of 1 to 1.5% by weight of said products. A weight gain of 15 to 30% was noted compared to a lot of control rats that were not given any protein concentrate.

### TEST 3:

A batch of chicks was fed with a food product consisting of commercial feed to which protein concentrate according to the invention was added in the proportion of 1.5% by weight. An average weight gain in the lot of chicks of 19% and a reduction of 75% in mortality was observed, as compared to a control lot, which was not given any protein concentrate.

### TEST 4:

A protein powder obtained in accordance with the invention is mixed with mineral compounds and oligo-elements in a proportion of 1.5 to 2% and the product obtained was administered to various animals. The following results were noted:

- when these products are administered to livestock and slaughterhouse calves, there is a general improvement in the general condition and a drop in the mortality rate, and, specifically, a weight gain of 3 to 5%;
- milk cows that were fed these powders show a better general condition, an improvement in the digestibility of feed, regularity of lactation and an increase in butyric levels;
- the quality of the meat of slaughterhouse cattle is improved when these powders are administered to these cattle;
- in piglets, the administration of these powders increases the speed of growth and their resistance to disease; when a protein powder according to the invention is administered to young sows in a proportion of 1% of the feed comprising their normal diet, there is a significant acceleration in the onset of puberty

**TEST 5:**

A powder prepared according to the invention is administered to trout in a proportion of 5 to 10% of their daily dry ration. Compared to a control group of trout to which no protein concentrate was administered, there is a noticeable increase in the rate of growth and a reduction in the mortality rate of these trout.



## CLAIMS

1. Process for the manufacturing of fish protein concentrates by enzymatic means using the proteolytic enzymes contained in the fish viscera, characterized in that a certain quantity of water is added to the raw material consisting of fish and/or fish viscera, in that the reaction mixture obtained is quickly raised to a temperature of approximately 40°C, in that the pH of the reaction mixture is brought to a level of around 4.5, in that the temperature of the medium is slowly brought to a temperature equal to a maximum of 70°C for a period such that a predetermined fraction of fish proteins is dissolved, these three stages constituting the enzymatic digestion properly so-called, and in that the liquid obtained at the end of digestion is treated in a suitable separation installation, in order to separate the fish oils and fats and the sediments comprised of undissolved substances and undigested proteins from a proteolysis juice rich in amino acids.

2. The process of Claim 1, characterized in that the first phase of rapid temperature increase up to 40°C takes place in around 30 to 45 minutes.

3. The process of Claim 1, characterized in that the second phase of slow temperature increase up to around 70°C takes around 4 to 10 hours.

4. The process of Claim 1, characterized in that the adjustment of the pH of the reaction medium to around 4.5 is done through the addition of any acid that does not affect the natural scale of the amino acids, preferably hydrochloric acid, acetic acid or lactic acid.

5. The process of Claim 1, characterized in that the water is added in a volume substantially equal to one fourth the volume of the raw material.

6. The process of one of the preceding claims, characterized in that during the enzymatic treatment, the reaction medium is agitated continuously by means of an agitator preferably performing at between twenty and twenty-five beats or rotations per minute.

7. The installation for carrying out the process of one of the preceding claims, characterized in that it includes a digester equipped with an agitator in which the digestion is carried out, a first centrifugal separator mounted following the digester to separate the sediments from the liquid containing the fish oils and fats and the proteolysis juice, a second centrifugal separator downstream from the first separator to receive this liquid and separate the fish oils and fats from the proteolysis juice, at least one proteolysis juice treatment device which substantially reduces the water content, and a sediment treatment unit; the proteolysis juice obtained on output from the second centrifugal separator can be conveyed by a first branch conveyor to the proteolysis juice treatment device or devices and/or a second branch conveyor to the intake of the sediment treatment unit.

8. The installation of Claim 7, characterized in that the first centrifugal separator rotates with an acceleration advantageously ranging between 2,000 and 2,200 G.

9. The installation of Claim 7, characterized in that the second centrifugal separator rotates with an acceleration advantageously ranging between 15,000 and 18,000 G.

10. The installation of Claim 7, characterized in that the sediment treatment unit consists of a rotary drier brought to a temperature of 130-140°C. then of a cooler that allows the product coming out of the rotary drier to be very quickly cooled, a grinder, a screening device and a bagging machine.

11. The installation of Claim 7, characterized in that the digester, which may be horizontal or vertical, is equipped with a rotary or alternating agitator, which preferably carried out twenty to twenty-five rotations or beats per minute.

12. A protein concentrate characterized in that it is made by the process according to any one of Claims 1 to 6.

PL. 1, 1

[See original for diagram]

Raw materials

Grinder

Digester

Pump

Liquids

Centrifugal  
separator

Sediments

Centrifugal  
separator

Pump

Rotary  
drier

Autolysis juice

Pasteurizer

Spray

Evaporator

Cooler

Grinder

Screening unit

Bagging unit

Oil  
decanting

for  
transformation

Storage  
Sale

Packaging and sale

Sale

**Pl. 11.2**

**Fig. 2**

[See original for drawing]